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AUTOMATED TRACE METALS ANALYZER

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ABSTRACT

Metal contamination arises from many sources and does not biodegrade in the environment. Prevention of soil and sediment contamination can only be accomplished by effective real or near-real-time monitoring of effluents, process streams, and runoff.

Field measurement of metals is difficult due to the size and complexity of the standard analytical instrumentation. Current laboratory methods employ either atomic absorption spectroscopy or inductively coupled plasma spectroscopy . Although these instruments are capable of low parts per billion measurements, they are expensive, sensitive to matrix affects, and require experienced operators as well as significant infrastructure support for field use. These systems are also not well suited for continuous automated monitoring of effluent and process streams.

A prototype automated trace metals analyzer has been developed based on the electrochemical technique of potentiometric stripping analysis. This technique was pioneered by D. Jagner in the early 70's. Recent advances in electronics now enable this technique to perform a completely automated metal analyses to less than one part per billion in about three minutes. The instrument, weighing less that 10 kg, provides a capability to perform on-site, discrete analyses, or continuous, automatic monitoring at programmed intervals over extended periods. The analyzer can be packaged in many configurations to suit specific requirements. These include minimum size for maximum portability, NEMA enclosures for industrial locations, or bench top systems for laboratory settings.

1. INTRODUCTION

In industrial process control and environmental compliance monitoring graphite furnace atomic absorption (AA) spectroscopy and inductively coupled plasma mass spectroscopy have been traditionally used to measure trace metals. These instruments are large, expensive, and require a high level of infrastructure support. Because of this, trace metal measurement usually involves sampling, preservation, and transport to a centralized laboratory for later analysis. Current field tests for metals are difficult to use under field and industrial conditions, usually lack simultaneous multianalyte capability, and require operator intervention. The Automated Trace Metals Analyzer (ATMA) overcomes most of these problems. This instrument is capable of automated multianalyte trace metal analysis in the field using potentiometric stripping analysis (PSA). This technique is capable of measuring over 40 metals with enough sensitivity to detect concentrations in the low parts per billion range[1]. It has been used to detect metals in diverse fluids such as drinking water[2], wine[3], sediment[4], and blood[5].

The windows based interface enable nontechnical personnel to setup the instrument and collect data with minimal training. The instrument continually monitors performance and will automatically notify the

operator and log any problems that it can not correct. While in operation the instrument is sealed from the external environment excluding the possibility of sample contamination.

2. PRINCIPLE OF OPERATION

The two step PSA process was originally developed by Jagner[6]. In the first step metals in the sample solution are reduced by applying a negative potential to the working electrode in a process similar to that occurring in a plating bath. This step concentrates the metals and typically requires about 30 seconds depending on the amount of metals in solution. The second step involves applying a constant current to the working electrode or removing the voltage potential from the working electrode while measuring the potential as a function of time. As the potential falls, each metal is oxidized at a specific voltage. During the metal oxidation the potential remains constant resulting in a plateau in the potential versus time plot (Figure 1.). The length of the plateau is proportional to the amount of metal originally in solution. To calculate the true concentration of the metal a standard curve is generated by adding known quantities of the metals of interest to the test sample and repeating the measurement. This entire process is totally automated and proceeds without any intervention. When the concentrations of the metals have been determined (usually in less than 3 minutes) the instrument displays and logs the concentrations and the relevant measurement statistics, pumps out the test sample, rinses the cell, and waits for either an operator command or the next programmed sampling interval.
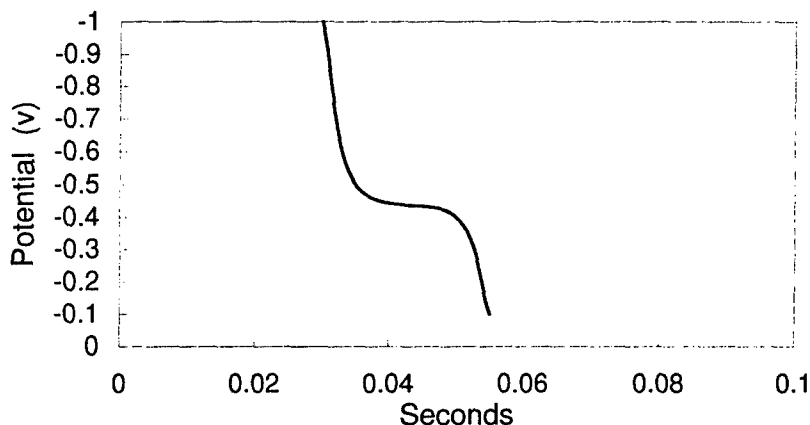

Figure 1. Potential of the reference electrode over time after the reducing voltage is removed. The potential falls until it reaches a value where a specific metal will be oxidized, in this case lead. The potential remains constant until all of the metal is oxidized. This plateau is proportional to the original concentration of metal in the solution. Each metal has a characteristic voltage at which it is oxidized enabling multiple metals to be measure simultaneously.

3. SYSTEM DESCRIPTION AND PERFORMANCE

The ATMA consists of two main components. The first component consists of a computer containing custom software and hardware used to control the electrochemical cell operations, data acquisition, and data analysis. The second component is the mechanical package holding the pumps, reagents, and test cell. The test cell contains a removable reference, counter, and working electrode. Injection ports are located radially around the cell which has a sample volume of 2.5 mL. The entire unit weighs less than 10 kg and can be transported in two cases which will fit under an airline seat.

The ATMA is capable of running unattended for up to ten days. The auto-sampling rate is adjustable up to a maximum of one sample per minute. The instrument can also be run manually for discrete

measurements under operator control or it can be triggered by an external signal from a user provided sensor.

A comparison of graphite furnace AA and ATMA results for lead levels in drinking water are shown in Figure 2. The samples were collected, split, and analyzed simultaneously with the AA and ATMA. The correlation coefficient (R^2) is 0.94 with a slope of 0.95 indicating agreement within experimental error between the two instruments.
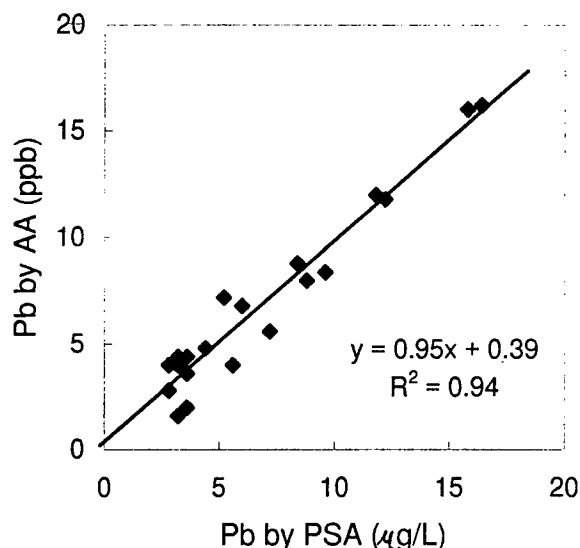
Figure 2. A comparison of graphite furnace AA and ATMA results for lead levels in drinking water. Each point is a single drinking water sample, the X axis is the level measured by the ATMA the Y axis is the value measured by the AA.
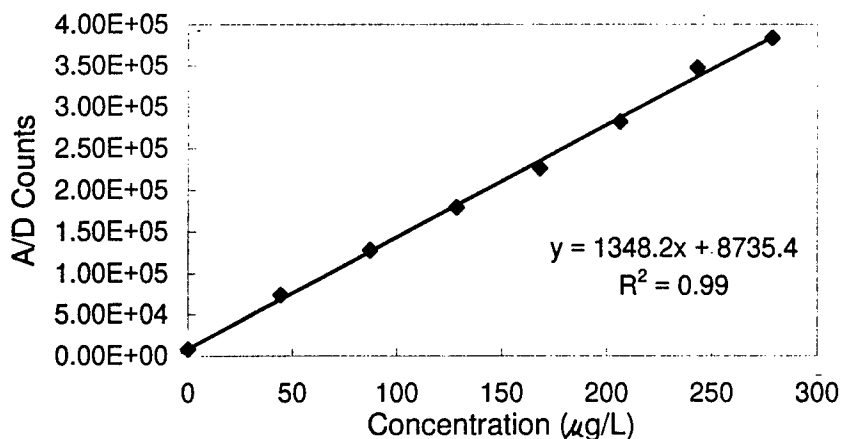
Figure 3. Linearity of the ATMA for lead concentrations between 0 and 300 μ g/L. A/D counts is the number of samples the instrument's A/D converter measured while the electrode potential was at the characteristic stripping potential for oxidizing lead.

The linearity of the ATMA over a range of 0 to 300 μ g/L lead is shown in Figure 3. An eight point standard addition test was conducted using a single sample of double distilled water spiked up to a level of 280 μ g/L. On the Y axis A/D counts is the number of samples the analog to digital converter

measured which were at the characteristic potential for oxidizing lead. This is a direct measurement of the length of the plateau shown in Figure 1. A correlation coefficient of greater than 0.99 was obtained indicating excellent linearity within this range.

4.CONCLUSIONS

The ATMA is useful in many situations where there is a need to know, with single parts per billion accuracy, the concentration of heavy metals in the field and in industrial process control. Because the instrument can be brought to the site for measurements the risk of sample contamination is much lower than with conventional on-site sample preservation and transport, in addition the automated capabilities allow completely unattended operation triggered at timed intervals or external events. Demonstrations under the Environmental Security Technology Certification Program have shown that the per sample cost is less than half that of conventional technology. This easy to use instrument with it's windows based operating system is a viable alternative to the expensive and complex laboratory equipment currently in use today.

5.ACKNOWLEDGMENTS

6.REFERENCES

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